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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

:

YOSHIKAZU OGURA, ET AL.

: EXAMINER: WEIER, A.J.

SERIAL NO: 10/532,727

:

FILED: APRIL 27, 2005

: ART UNIT: 1794

FOR: METHOD OF REMOVING CAFFEINE FROM CAFFEINE-CONTAINING
CATECHIN COMPOUND COMPOSITIONDECLARATION UNDER 37 C.F.R. § 1.132COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes Masahiro Fukuda who deposes and states that:

1. I am a graduate of Tokyo University of Technology and Agriculture and received my Master degree in the field of chemistry, in the year 1986.
2. I have been employed by KAO corporation, for 20 years in the field of Research & Development.
3. I understand the English language or, at least, that the contents of the Declaration were made clear to me prior to executing the same.
4. The following experiments were carried out by me or under my direct supervision and control.

5. The following experiment was performed to demonstrate the criticality of the weight ratio of organic solvent to water of no more than 9/1.

6. The experiments in this Declaration were conducted according to the following protocol:

Comparative Example 10

The caffeine-containing catechin composition ("POLYPHENONE HG", product of Tokyo Food Techno Co., Ltd.; 100 g) was suspended in 99.5 % EtOH (900 g) at room temperature under stirring at 250 rpm, and subsequent to the addition of activated carbon ("KURARAY COAL GLC", product of Kuraray Chemical K.K.; 20 g), stirring was continued for about 20 minutes. Subsequently, stirring was continued for about 30 minutes still at room temperature. Subsequent to filtration through No. 2 filter paper, re-filtration was conducted through a 0.2- μ m membrane filter. Finally, until the water content was reduced to a similar level as in Example 1, water was caused to gradually evaporate in a drier to afford a product.

Comparative Example 11

The caffeine-containing catechin composition ("POLYPHENONE HG", product of Tokyo Food Techno Co., Ltd.; 100 g) was suspended in a 99.5% aqueous solution of ethanol (900 g) at room temperature under stirring at 250 rpm, and subsequent to the addition of activated carbon ("KURARAY COAL GLC", product of Kuraray Chemical K.K.; 30 g) and acid clay ("MIZUKA ACE #600", product of Mizusawa Chemical Industries, Ltd.; 30g), stirring was continued for about 20 minutes. Subsequently, stirring was continued for about 30 minutes still at room temperature. Subsequent to filtration through No. 2 filter paper, re-

filtration was conducted through a 0.2- μ m membrane filter. Finally, until the water content was reduced to a similar level as in Example 2, water was gradually evaporated in a drier to afford a product.

7. The results are shown in the following tables:

	Comp. Ex. 10
Caffeine-containing catechin composition (g) ("POLYPHENONE HG", product to Tokyo Food Techno Co., Ltd)	100
99.5 %Ethanol (g)	900
Water (g)	0
Activated carbon (g) ("KURARAY COAL GLC", product of Kuraray Chemical K.K.)	20
Organic solvent/water (weight ratio)	99.5/0.5
Amounts of catechins after removal of ethanol (wt%)	
GC (gallocatechin)	6.89
EGC (epigallocatechin)	32.4
C (catechin)	2.03
EC (epicatechin)	9.12
EGCg (epigallocatechin gallate)	37.0
GCg (gallocatechin gallate)	1.39
ECg (epicatechin gallate)	10.4
Cg (catechin gallate)	0.70
Amount of catechins after purification/amount of catechins before purification	1.037
Concentration of caffeine after purification (mg%)	27.1
Amount of caffeine after purification/Amount of caffeine before purification [-]	0.741
Non-polymer catechins after purification/caffeine after purification [-]	7.0
Content of gallates in non-polymer catechins after treatment (wt.%)	49.5
Absorbance (-)	0.075
Evaluation of purified product	a bitter taste was still existed.

Application No. 10/532,727
 Declaration under 37 C.F.R. § 1.132

	Comp. Ex. 11
Caffeine-containing catechin composition (g) ("POLYPHENONE HG", product to Tokyo Food Techno Co., Ltd)	100
99.5% Ethanol (g)	900
Water (g)	0
Activated carbon (g) ("KURARAY COAL GLC", product of Kuraray Chemical K.K.)	30
Acid clay (g) ("MIZUKA ACE #600", product of Mizusawa Chemical Industries, Ltd.)	30
Organic solvent/water (weight ratio)	99.5/0.5
Amounts of non-polymer catechins after treatment (wt%)	
GC (gallocatechin)	6.91
EGC (epigallocatechin)	32.41
C (catechin)	1.98
EC (epicatechin)	8.91
EGCg (epigallocatechin gallate)	36.64
GCg (gallocatechin gallate)	1.52
ECg (epicatechin gallate)	11.01
Cg (catechin gallate)	0.71
Non-polymer catechins/caffeine after treatment (weight ratio)	7.9
Amount of caffeine after treatment (mg/100 mL)	16.2
Content of gallates in non-polymer catechins after treatment (wt%)	49.8
Content of gallocatechins in non-polymer catechins after treatment (wt%)	76.0
Absorbance (-)	0.151
Evaluation of purified product	Hue was deteriorated, and a bitter taste was still existed.

8. Comparative Example 10 shows the experimental example of a process wherein a caffeine-containing catechin composition is dissolved in a mixed solution whose weight ratio of organic solvent to water is greater than 9/1 and then treated with activated carbon. The product thus obtained has been found to suffer from persistent bitterness.

Application No. 10/532,727
Declaration under 37 C.F.R. § 1.132

Comparative Example 11 shows the experimental example of a process wherein a caffeine-containing catechin composition is dissolved in a mixed solution whose weight ratio of organic solvent to water is greater than 9/1 and then treated with activated carbon and acid clay. The product thus obtained has been found to have deteriorating color tone and also suffer from persistent bitterness.

As can be understood from the combined evidence of Examples 1-4 and Comparative Examples 1-4 of the present application and the above additional data Comparative Examples 10 and 11, it has been confirmed that caffeine can be removed selectively by adjusting the weight ratio of organic solvent to water, each of which is contained in the mixed solution in which the caffeine-containing catechin composition is dissolved, to a range of from 9/1 to 1/9. What is more, it is evident that the claimed process is greatly effective at obtaining a catechin composition having many advantages, such as the prevention of precipitates, improved color tone and excellently good taste.

9. I declare further that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

10. Further Declarant saith not

Masahiro Fukuda
Name:

January 16, 2009
Date